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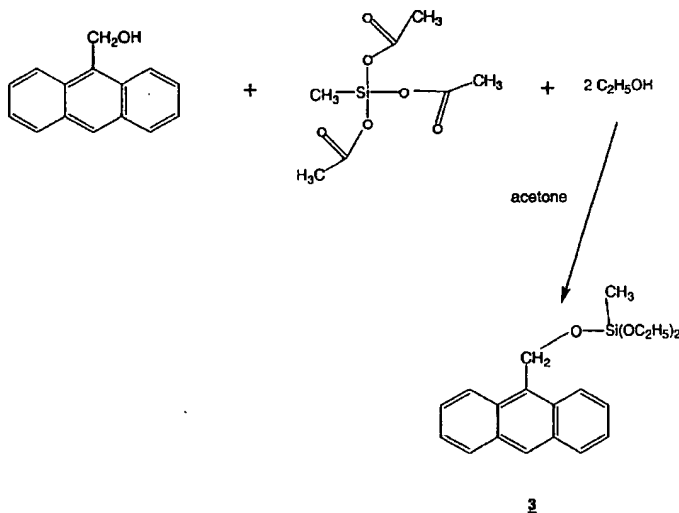
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(54) Title: ABSORBING COMPOUNDS FOR SPIN-ON GLASS ANTI-REFLECTIVE COATINGS FOR PHOTOLITHOGRAPHY



(57) Abstract: An absorbing ether-like compound including a siliconethoxy, silicondiethoxy, or silicontriethoxy species attached to a naphthalene or anthracene chromophore via an oxygen linkage is used as an organic light-absorbing compound. The absorbing ether-like compound is incorporated into spin-on glass materials to provide anti-reflective coating materials for deep ultraviolet photolithography. A method of synthesizing the light-absorbing ether compounds is based on the reaction of an alcohol-substituted chromophore with an acetoxysilicon compound in the presence of alcohol. A method of making absorbing spin-on-glass materials including the absorbing ether-like compounds is also provided.



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**ABSORBING COMPOUNDS FOR SPIN-ON-GLASS  
ANTI-REFLECTIVE COATINGS FOR PHOTOLITHOGRAPHY**

**TECHNICAL FIELD**

5           The present invention relates generally to light-absorbing spin-on glass materials and more specifically to absorbing compounds that may be incorporated in spin-on glass materials for use as anti-reflective layers in photolithography and methods of producing the absorbing compounds.

**BACKGROUND**

10           To meet the requirements for faster performance, the characteristic dimensions of features of integrated circuit devices have continued to be decreased. Manufacturing of devices with smaller feature sizes introduces new challenges in many of the processes conventionally used in semiconductor fabrication. One of the most important of these fabrication processes is photolithography.

15           It has long been recognized that linewidth variations in patterns produced by photolithography can result from optical interference from light reflecting off an underlying layer on a semiconductor wafer. Variations in photoresist thickness due to the topography of the underlying layer also induce linewidth variations. Anti-reflective coatings (ARC) applied under a photoresist layer have been used to prevent interference from reflection of the  
20   irradiating beam. In addition, anti-reflective coatings partially planarize the wafer topography, helping to improve linewidth variation over steps because the photoresist thickness is more uniform.

          Organic polymer films, particularly those that absorb at the i-line (365 nm) and g-line (436 nm) wavelengths conventionally used to expose photoresists, and at the recently used  
25   248 nm wavelength, have been employed as anti-reflective coatings. However, the fact that the organic ARC's share many chemical properties with the organic photoresists can limit usable process sequences. Furthermore organic ARC's may intermix with photoresist layers. One solution to avoid intermixing, is to introduce thermosetting binders as additional components of organic ARC's, as described, for example in U.S. Patent No. 5,693,691 to  
30   Flaim et al. Dyes may also be incorporated in organic ARC's, as well as, optionally,

additional additives such as wetting agents, adhesions promoters, preservatives, and plasticizers, as described in U. S. Patent No. 4,910,122 to Arnold et al.

Silicon oxynitride is another material that has been used as an anti-reflective coating. However, silicon oxynitride works as an ARC by a destructive interference process rather than by absorption, which means that very tight control of the oxynitride thickness is  
5 necessary and that the material may not work well as an ARC over highly variable topography. Furthermore silicon oxynitride is typically deposited by chemical vapor deposition, while photoresist layers are typically applied using a spin-coater. The additional chemical vapor deposition process can add to processing complexity.

10 Yet another class of materials that can be used as an anti-reflective layer is spin-on-glass (SOG) compositions containing a dye. Yau et al., U.S. Patent No. 4,587,138, disclose a dye such as basic yellow #11 mixed with a spin-on-glass in an amount approximately 1% by weight. Allman et al. U. S. Patent No. 5,100,503 disclose a cross-linked polyorganosiloxane containing an inorganic dye such as  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_7$ ,  $\text{MoO}_4$ ,  $\text{MnO}_4$ , or  $\text{ScO}_4$ , and an adhesion  
15 promoter. Allman additionally teaches that the spin-on-glass compositions also serve as a planarizing layer. However, the spin-on-glass, dye combinations that have been disclosed to date are not optimal for exposure to the deep ultraviolet, particularly 248 and 193 nm, light sources that are coming into use to produce devices with small feature sizes. In addition, not all dyes can be readily incorporated into an arbitrary spin-on-glass composition.

20 Thus there remains a need for compounds absorbing strongly in the deep ultraviolet spectral region that may be incorporated into spin-on-glass compositions to provide anti-reflective coatings and for methods of synthesizing such absorbing compounds.

#### SUMMARY

An anti-reflective coating material for deep ultraviolet photolithography includes one  
25 or more organic absorbing compounds incorporated into a spin-on-glass (SOG) material. According to an embodiment of the present invention, an absorbing ether-like compound including a siliconethoxy, silicondiethoxy, or silicontriethoxy species attached to a naphthalene or anthracene chromophore via an oxygen linkage is used as an organic absorbing compound. The absorbing ether-like compounds have a general formula  
30  $\text{C}_{14}\text{H}_9(\text{CH}_2)_n\text{OSiR}_m(\text{OC}_2\text{H}_5)_{3-m}$  or  $\text{C}_{10}\text{H}_8(\text{CH}_2)_n\text{OSiR}_m(\text{OC}_2\text{H}_5)_{3-m}$ , where  $n = 1-3$ ,  $m=0-2$ , and R is hydrogen, or an alkyl group such as a methyl, ethyl, or propyl group.

A method of synthesizing the light-absorbing ether-like compounds of the present invention is based on the reaction of an alcohol-substituted chromophore with an acetoxysilicon compound of the general formula  $R_mSi(OCOCH_3)_{4-m}$ , in the presence of a stoichiometric amount of alcohol, where the reactants have the molar ratio of 1:1:3-m. For example, the synthesis of 9-anthracene methoxy-methyldiethoxysilane uses 9-anthracene methanol, methyltriacetoxysilane (MTAS), and ethanol in a molar ratio of 1:1:2 as reactants. The reactants are combined with acetone, or a suitable alternative ketone, to form a reaction mixture which is stirred for an extended period sufficient to form the product, and then the acetic acid byproduct is removed by inert gas purging or by vacuum extraction.

The absorbing ether-like compounds may be incorporated into spin-on-glass materials including methylsiloxane, methylsilsesquioxane, phenylsiloxane, phenylsilsesquioxane, methylphenylsiloxane, methylphenylsilsesquioxane, and silicate polymers. As used herein, spin-on-glass materials also include hydrogensiloxane polymers of the general formula  $(H_{0.1-1.0}SiO_{1.5-2.0})_x$  and hydrogensilsesquioxane polymers, which have the formula  $(HSiO_{1.5})_x$ , where x is greater than about 8. Also included are copolymers of hydrogensilsesquioxane and alkoxyhydridosiloxane or hydroxyhydridosiloxane. Spin-on-glass materials additionally include organohydridosiloxane polymers of the general formula  $(H_{0.1-1.0}SiO_{1.5-2.0})_n(R'_{0.1-1.0}SiO_{1.5-2.0})_m$ , and organohydridosilsesquioxane polymers of the general formula  $(HSiO_{1.5})_n(R'SiO_{1.5})_m$ , where m is greater than 0 and the sum of n and m is greater than about 8 and R' is alkyl or aryl. Coating solutions of spin-on-glass materials incorporating absorbing compounds are used to form anti-reflecting films on various layers in integrated circuit devices.

According to another aspect of the present invention, methods for synthesizing absorbing spin-on-glass compositions including the absorbing ether compounds are also provided.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 depicts the reaction scheme for synthesizing 9-anthracene methoxy-methyldiethoxysilane using methyltriacetoxysilane, according to an embodiment of the present invention.

## DETAILED DESCRIPTION

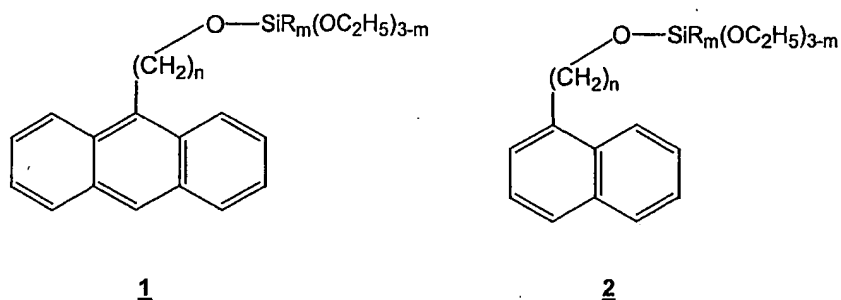
An anti-reflective coating material for deep ultraviolet photolithography includes one or more organic absorbing compounds incorporated into a spin-on-glass (SOG) material. The absorbing spin-on-glass compositions are dissolved in appropriate solvents to form coating solutions and applied to various layers of materials in fabricating semiconductor devices. The absorbing spin-on-glass anti-reflective coatings have been designed to be readily integrated into existing semiconductor fabrication processes. Properties that provide integration include developer resistance, thermal stability during standard photoresist processing, and selective removal with respect to underlying layers.

Many naphthalene- and anthracene-based compounds have significant absorption at 248 nm and below. Benzene-based, equivalently termed here phenyl-based, compounds have significant absorption at wavelengths shorter than 200 nm. While these naphthalene-, anthracene-, and phenyl-based compounds are frequently referred to as dyes, the term absorbing compound is used here because the absorptions of these compounds are not limited to wavelengths in the visible region of the spectrum. However, not all such absorbing compounds can be incorporated into spin-on-glasses for use as ARC materials. Absorbing compounds suitable for use with the present invention have an absorption peak over at least an approximately 10 nm wide wavelength range around wavelengths such as 248 nm, 193 nm, or other ultraviolet wavelengths, such as 365 nm, that may be used in photolithography. Absorbing compounds which only have narrow absorption peaks, for example, less than 2 nm wide, around these wavelengths are not as desirable.

The chromophores of suitable absorbing compounds typically have one, two, or three benzene rings that may or may not be fused. Incorporatable absorbing compounds have an accessible reactive group attached to the chromophore, the reactive groups including hydroxyl groups, amine groups, carboxylic acid groups, and substituted silyl groups with silicon bonded to one, two, or three "leaving groups," such as alkoxy groups or halogen atoms. Ethoxy or methoxy groups or chlorine atoms are frequently used as leaving groups. Thus, suitable reactive groups include siliconethoxy, silcondiethoxy, silcontriethoxy, siliconmethoxy, silcondimethoxy, silcontrimethoxy, chlorosilyl, dichlorosilyl, and trichlorosilyl groups. Specific examples of incorporatable absorbing compounds for spin-on glass compositions are described in U.S. Patent Application Serial No. 09/491,166, which is commonly assigned with the present application and incorporated herein by reference. The

inclusion of reactive groups with one or more ethoxy groups bonded to silicon has been found to be advantageous, especially for promoting thermal stability of the absorbing SOG films.

According to an aspect of the present invention, an incorporatable absorbing  
 5 compound includes a siliconethoxy, silcondiethoxy, or silcontriethoxy species as a reactive group attached to a naphthalene or anthracene chromophore via an oxygen linkage. Thus, the absorbing compounds of the present invention are ether-like. (If the silicon atom in the absorbing compound were replaced by a carbon atom, the compounds would be strictly classified as ethers.) The compounds may have the general structure



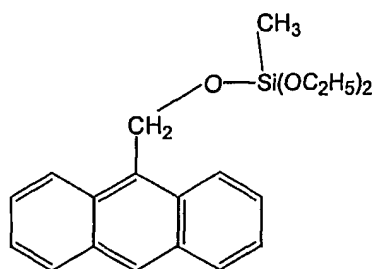
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where  $n = 1-3$ ,  $m=0-2$ , and R is hydrogen or an alkyl group such as a methyl, ethyl, or propyl group. Structures **1** and **2** illustrate compounds in which the reactive group is attached to the chromophore at a particular substituent site. Analogous compounds in which reactive groups are attached to alternative sites, all described by the general formulas

15  $C_{14}H_9(CH_2)_nOSiR_m(OC_2H_5)_{3-m}$  or  $C_{10}H_8(CH_2)_nOSiR_m(OC_2H_5)_{3-m}$  are also included in the present invention.

A method of synthesizing the light-absorbing ether-like compounds of structures **1** and **2** is based on the reaction of an alcohol-substituted fused ring chromophore with an acetoxysilicon compound in the presence of alcohol. The acetoxysilicon reactant is given by  
 20 the general formula  $R_mSi(OCOCH_3)_{4-m}$ , where R and m are defined above. Particular examples of useful acetoxysilicon compounds include methyltriacetoxysilane (MTAS), tetraacetoxysilane (TAS), dimethyldiacetoxysilane, and diethyldiacetoxysilane. Useful alcohols include methanol, ethanol, and propanol.

For example the synthesis of 9-anthracene methoxy-methyldiethoxysilane **3**

**3**

uses 9-anthracene methanol, MTAS, and ethanol in a molar ratio of 1:1:2 as reactants. As can be understood from the reaction mechanism of Fig. 1, one acetoxy group on MTAS reacts with the  $-\text{CH}_2\text{OH}$  on 9-anthracene methanol to form the ether linkage to the silicon-containing reactive group and acetic acid while two acetoxy groups on MTAS react with ethanol to form the ethoxy group and acetic acid. In the synthesis method, the reactants are combined with acetone, or an alternative ketone such as methylisobutylketone (MIBK) or methylethylketone, to form a reaction mixture which is stirred for an extended period sufficient to form the product **3**, typically several days, and then the acetic acid byproduct is removed by purging with inert gas or by vacuum extraction. From this reaction mechanism, it may be understood that in general, the ratio of alcohol-substituted fused ring compound to acetoxysilicon compound,  $\text{R}_m\text{Si}(\text{OCOCH}_3)_{4-m}$ , to alcohol is 1:1:3-m.

The absorbing ether-like compounds may be incorporated in spin-on-glass materials including methylsiloxane, methylsilsesquioxane, phenylsiloxane, phenylsilsesquioxane, methylphenylsiloxane, methylphenylsilsesquioxane, and silicate polymers. As used herein, spin-on-glass materials also include hydrogensiloxane polymers of the general formula  $(\text{H}_{0-1.0}\text{SiO}_{1.5-2.0})_x$  and hydrogensilsesquioxane polymers, which have the formula  $(\text{HSiO}_{1.5})_x$ , where x is greater than about 8. Also included are copolymers of hydrogensilsesquioxane and an alkoxyhydridosiloxane or hydroxyhydridosiloxane. Spin-on-glass materials additionally include organohydridosiloxane polymers of the general formula  $(\text{H}_{0-1.0}\text{SiO}_{1.5-2.0})_n(\text{R}'_{0-1.0}\text{SiO}_{1.5-2.0})_m$ , and organohydridosilsesquioxane polymers of the general formula  $(\text{HSiO}_{1.5})_n(\text{R}'\text{SiO}_{1.5})_m$ , where m is greater than 0 and the sum of n and m is greater than about 8 and R' is alkyl or aryl. Some useful organohydridosiloxane polymers have the sum of n and m from about 8 to about 5000 where R' is a  $\text{C}_1$ - $\text{C}_{20}$  alkyl group or a  $\text{C}_6$ - $\text{C}_{12}$  aryl group. The organohydridosiloxane and organohydridosilsesquioxane polymers are alternatively denoted spin-on-polymers. Specific examples include methylhydridosiloxanes,



ethylhydridosiloxanes, propylhydridosiloxanes, t-butylhydridosiloxanes, phenylhydridosiloxanes, methylhydridosilsesquioxanes, ethylhydridosilsesquioxanes, propylhydridosilsesquioxanes, t-butylhydridosilsequioxanes, phenylhydridosilsesquioxanes, and combinations, thereof.

5 In the absorbing spin-on-glass, compositions, the absorbing compounds may be incorporated interstitially in the spin-on-glass matrix. Alternatively, the absorbing compounds are chemically bonded to the spin-on-glass polymer. Without being bound to any theory, the inventors suggest that bonding of incorporatable absorbing compounds to the spin-on-glass polymer backbone via the accessible reactive groups provides beneficial  
10 results.

Spin-on-glass materials are typically synthesized from a variety of silane reactants including, for example, triethoxysilane (HTEOS), tetraethoxysilane (TEOS), methyltriethoxysilane (MTEOS), dimethyldiethoxysilane, dimethyldimethoxysilane, tetramethoxysilane (TMOS), methyltrimethoxysilane (MTMOS), trimethoxysilane,  
15 dimethyldimethoxysilane, phenyltriethoxysilane (PTEOS), phenyltrimethoxysilane (PTMOS), diphenyldiethoxysilane, and diphenyldimethoxysilane. Halosilanes, particularly chlorosilanes such as trichlorosilane, methyltrichlorosilane, ethyltrichlorosilane, phenyltrichlorosilane, tetrachlorosilane, dichlorosilane, methyldichlorosilane, dimethyldichlorosilane, chlorotriethoxysilane, chlorotrimethoxysilane,  
20 chloromethyltriethoxysilane, chloroethyltriethoxysilane, chlorophenyltriethoxysilane, chloromethyltrimethoxysilane, chloroethyltrimethoxysilane, and chlorophenyltrimethoxysilane are also used as silane reactants. To produce absorbing spin-on-glass compositions, the absorbing ether compounds, such as structures (1) or (2), or combinations of structures (1) and/or (2) and other absorbing compounds, are combined with  
25 the silane reactants during the synthesis of the SOG materials.

In a typical method of producing an absorbing spin-on glass composition, a reaction mixture including silane reactants, for example HTEOS, or TEOS and MTEOS, or, TMOS and MTMOS; or, alternatively, tetrachlorosilane and methyltrichlorosilane, one or more absorbing compounds; a solvent or combination of solvents; and an acid/water mixture, is  
30 formed in a reaction vessel. Appropriate solvents include acetone, 2-propanol, and other simple alcohols, ketones and esters such as 1-propanol, MIBK, propoxypropanol, and propyl acetate. The acid/water mixture is, for example nitric acid and water. Other protic acids or

acid anhydrides, such as acetic acid, formic acid, phosphoric acid, hydrochloric acid or acetic anhydride are alternatively used in the acid mixture. The resulting mixture is held at a temperature between about 30 and 80 °C for between approximately 1 and 24 hours to produce the absorbing SOG polymer solution.

5           The absorbing SOG can be diluted with appropriate solvents to achieve coating solutions that produce films of various thicknesses. Suitable dilutant solvents include acetone, 2-propanol, ethanol, butanol, methanol, propylacetate, ethyl lactate, propylene glycol methyl ether acetate, and propylene glycol propyl ether, referred to commercially as Propasol-P. Dilutant solvents with high boiling points such as ethyl lactate and propylene  
10   glycol propyl ether have been found to be beneficial. It is believed high boiling point solvents decrease the probability of formation of bubble film defects. In contrast, lower boiling point solvents may become entrapped below a crosslinked top layer of a film and subsequently produce voids when driven off during a baking process step. Additional solvents useful in the invention include ethylene glycol dimethyl ether, alternatively termed  
15   glyme, anisole, dibutyl ether, dipropyl ether, and pentanol. Optionally, surfactants, such as the product FC430, provided by 3M (Minneapolis, MN), or the product Megaface R08, provided by DIC (Japan), are also added to the coating solution. The coating solution is typically between about 0.5 and 20 % polymer by weight. Prior to use, the coating solution is filtered by standard filtration techniques.

20           A method of forming an absorbing organohydridosiloxane material includes forming a mixture of a dual phase solvent which includes both a non-polar solvent and a polar solvent and a phase transfer catalyst; adding one or more organotrihalosilane, hydridotrihalosilane, and one or more of absorbing compounds, to provide a dual phase reaction mixture; and reacting the dual phase reaction mixture for between 1 and 24 hours to produce the absorbing  
25   organohydridosiloxane polymer. The phase transfer catalyst includes but is not limited to tetrabutylammonium chloride and benzyltrimethylammonium chloride. Exemplary non-polar solvents include, but are not limited to, pentane, hexane, heptane, cyclohexane, benzene, toluene, xylene, halogenated solvents such as carbon tetrachloride and mixtures thereof. Useful polar solvents include water, alcohols, and alcohol and water mixtures. The absorbing  
30   polymer solution is diluted and filtered as described above to form a coating solution.

          The absorbing SOG coating solutions are applied to various layers used in semiconductor processing, depending on the specific fabrication process, typically by

conventional spin-on deposition techniques. These techniques include a dispense spin, a thickness spin, and thermal bake steps, to produce an absorbing SOG anti-reflective film. Typical processes include a thickness spin of between 1000 and 4000 rpm for about 20 seconds and two or three bake steps at temperatures between 80°C and 300°C for about one  
5 minute each. As reported in Example 2 below, an absorbing SOG anti-reflective film with absorbing ether of structure (3) exhibits an extinction coefficient greater than 0.18.

The methods of synthesizing 9-anthracene methoxy-methyldiethoxysilane **3**, 9-anthracene methoxy-triethoxysilane and the absorbing SOG material including these absorbing compounds are described in the following examples.

10

### **Example 1**

#### **Synthesis of 9-anthracene methoxy-methyldiethoxysilane**

In a 3 liter flask, 92.37 grams (0.419 moles) methyltriacetoxysilane (MTAS), 87.36 grams (0.419 moles) 9-anthracene methanol, 38.56 grams (0.839 moles) ethanol, 595.51 grams (10.20 moles) acetone were combined. The solution was stirred in a nitrogen  
15 atmosphere for 7 days. The solution was degassed to remove the acetic acid byproduct.

### **Example 2**

#### **Synthesis of absorbing SOG containing 9-anthracene methoxy-methyldiethoxysilane**

In a 1-liter flask 297 grams (4.798 moles), 2-Propanol, 148 grams (2.558 moles)  
20 acetone, 123 grams (0.593 moles) TEOS, 77 grams (0.432 moles) MTEOS, 200 g 9-anthracene methoxy-methyldiethoxysilane, produced in Example 1, 2.61 grams (0.009) moles rosolic acid, 10 grams (0.024 moles) 2-hydroxy-4(3-triethoxysilylpropoxy)-diphenylketone, 0.09 grams (0.0004 moles) anthraflavic acid, 0.6 grams 1.0 M nitric acid and 72 grams (3.716 moles) deionized water were combined. The flask was refluxed for 4 hours.  
25 To the solution, 43 grams (0.590 moles) of butanol was added. The solution was filtered. The solution was dispensed, followed by a 3000 rpm thickness spin for 20 seconds, and baked at 80°C and at 180°C for one minute each. Optical properties were measured with an N & K Technology Model 1200 analyzer. The film thickness was 2801 Å. At 248 nm, the refractive index (n) was 1.470 and the extinction coefficient (k) was 0.185.

**Example 3****Synthesis of 9-anthracene methoxy-triethoxysilane**

In a 3 liter flask, 110.73 grams (0.419 moles) tetra-acetoxysilane (TAS), 87.36 grams (0.419 moles) 9-anthracene methanol, 57.98 grams (1.2585 moles) ethanol, 595.51 grams (10.20 moles) acetone are combined. The solution is stirred in a nitrogen atmosphere for 7 days. The solution is degassed to remove the acetic acid byproduct.

**Example 4****Synthesis of absorbing SOG containing 9-anthracene methoxy-triethoxysilane**

In a 1-liter flask 297 grams (4.798 moles), 2-Propanol, 148 grams (2.558 moles) acetone, 123 grams (0.593 moles) TEOS, 77 grams (0.432 moles) MTEOS, 200 g 9-anthracene methoxy-triethoxysilane, produced in Example 3, 0.6 grams 1.0 M nitric acid and 72 grams (3.716 moles) deionized water are combined. The flask is refluxed for 4 hours. To the solution, 43 grams (0.590 moles) of butanol is added. The solution is filtered.

**Example 5****Synthesis of absorbing SOG containing 9-anthracene methoxy-methyldiethoxysilane**

In a 1-liter flask 297 grams (4.798 moles), 2-Propanol, 148 grams (2.558 moles) acetone, 123 grams (0.593 moles) TEOS, 77 grams (0.432 moles) MTEOS, 200 g 9-anthracene methoxy-methyldiethoxysilane, produced in Example 1, 0.6 grams 1.0 M nitric acid and 72 grams (3.716 moles) deionized water are combined. The flask is refluxed for 4 hours. To the solution, 43 grams (0.590 moles) of butanol is added. The solution is filtered.

Although the invention has been described with reference to particular examples, the description is only an example of the invention's application and should not be taken as a limitation. Various adaptations and combinations of features of the examples disclosed are within the scope of the invention as defined by the following claims.

### CLAIMS

We claim:

1. A method of making a light-absorbing ether-like compound, the method comprising:  
5 combining an alcohol-substituted fused benzene ring compound, the fused ring compound comprising two or three rings, an acetoxysilicon compound, an alcohol, and a solvent to produce a reaction mixture;  
stirring the reaction mixture for a time period sufficient to form the light-absorbing ether compound; and  
10 removing acidic byproducts.
2. The method of Claim 1 wherein the acetoxysilicon compound has the general formula  $R_mSi(OCOCH_3)_{4-m}$ , where R is selected from the group consisting of hydrogen, methyl, ethyl, and propyl, and  $m=0-2$ , and wherein the fused ring compound, the  
15 acetoxysilicon compound, and the alcohol are combined in the stoichiometric ratio of 1:1:3-m.
3. The method of Claim 2 wherein the acetoxysilicon compound is methyltriacetoxysilane.  
20
4. The method of Claim 2 wherein the fused ring compound is 9-anthracene methanol.
5. The method of Claim 2 wherein the alcohol is ethanol.  
25
6. The method of Claim 3 wherein the light-absorbing ether compound is 9-anthracene methoxy-methyldiethoxysilane.
7. A light-absorbing spin-on glass composition comprising a siloxane polymer  
30 and an absorbing ether-like compound comprising two or three fused benzene rings and an oxygen linkage to a silicon-containing species selected from the group consisting of siliconethoxy, silicondiethoxy, and silicontriethoxy.

8. The composition of Claim 7 wherein the absorbing ether compound has the formula  $C_{14}H_9(CH_2)_nOSiR_m(OC_2H_5)_{3-m}$  or  $C_{10}H_8(CH_2)_nOSiR_m(OC_2H_5)_{3-m}$ , wherein  $n=1-3$ ,  $m=0-2$ , and R is selected from the group consisting of hydrogen, methyl, ethyl, and propyl.
9. The composition of Claim 8 wherein the absorbing ether compound is 9-anthracene methoxy-methyldiethoxysilane.
10. The composition of Claim 7 wherein the siloxane polymer is a polymer selected from the group consisting of methylsiloxane, methylsilsesquioxane, phenylsiloxane, phenylsilsesquioxane, methylphenylsiloxane, methylphenylsilsesquioxane, and silicate polymers.
11. The composition of Claim 7 wherein the siloxane polymer is a polymer selected from the group consisting of hydrogensiloxane, hydrogensilsesquioxane, organohydridosiloxane, and organohydridosilsesquioxane polymers; and copolymers of hydrogensilsesquioxane and an alkoxyhydridosiloxane or hydroxyhydridosiloxane.
12. The composition of Claim 11 wherein the siloxane polymer is a polymer of a general formula selected from the group consisting of  $(H_{0-1.0}SiO_{1.5-2.0})_x$ , where x is greater than about 8, and  $(H_{0-1.0}SiO_{1.5-2.0})_n(R'_{0-1.0}SiO_{1.5-2.0})_m$ , where m is greater than 0, the sum of n and m is from about 8 to about 5000 and R' is a  $C_1-C_{20}$  alkyl group or a  $C_6-C_{12}$  aryl group.
13. A coating solution comprising the absorbing spin-on-glass composition of Claim 7 and a solvent or a solvent mixture.
14. The coating solution of Claim 13 wherein the solution is between about 0.5% and about 20% by weight absorbing spin-on-glass composition.
15. A method of making an absorbing spin-on-glass composition comprising: combining one or more silane reactants selected from the group consisting of alkoxysilanes and halosilanes, one or more incorporatable organic absorbing compounds, wherein at least one organic absorbing compound is an absorbing ether compound comprising two or three fused benzene rings and an oxygen linkage to a silicon-containing species selected from the group consisting of siliconethoxy,

silicondiethoxy, and silicontriethoxy, an acid/water mixture, and one or more solvents to form a reaction mixture; and

holding the reaction mixture at a temperature between about 30 and about 80 °C for a time sufficient to form the absorbing spin-on-glass composition.

- 5           16.    The method of Claim 15 wherein the one or more silane reactants are selected from the group consisting of triethoxysilane, tetraethoxysilane, methyltriethoxysilane, dimethyldiethoxysilane, dimethyldimethoxysilane, tetramethoxysilane, methyltrimethoxysilane, trimethoxysilane, dimethyldimethoxysilane, phenyltriethoxysilane, phenyltrimethoxysilane, diphenyldiethoxysilane, and diphenyldimethoxysilane,
- 10   trichlorosilane, methyltrichlorosilane, ethyltrichlorosilane, phenyltrichlorosilane, tetrachlorosilane, chlorotriethoxysilane, chlorotrimethoxysilane, chloromethyltriethoxysilane, chloroethyltriethoxysilane, chlorophenyltriethoxysilane, chloromethyltrimethoxysilane, chloroethyltrimethoxysilane, and chlorophenyltrimethoxysilane.
17.    The method of Claim 16 wherein the one or more silane reactants are
- 15   tetraethoxysilane and methyltriethoxysilane.
18.    The method of Claim 15 wherein the acid/water mixture is a nitric acid/water mixture.

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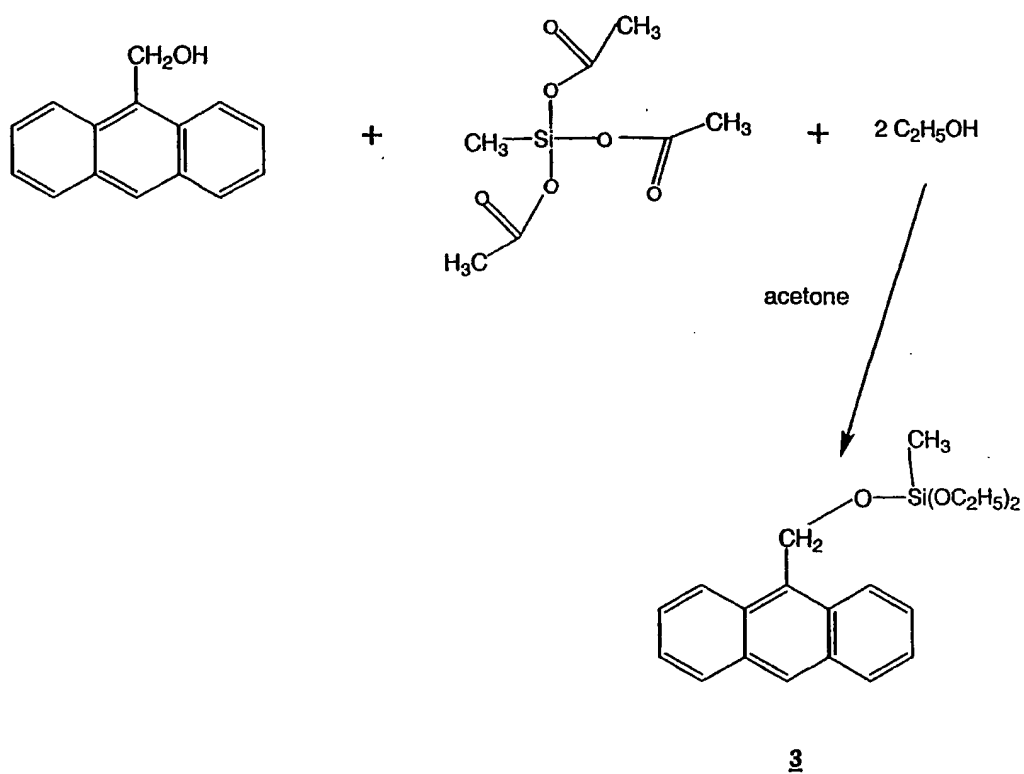


Fig. 1



# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US01/22232

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C08L 83/04

US CL : 556/458, 442; 528/34, 43, 39; 430/272.1; 525/477

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 556/458, 556/442, 528/34, 528/43, 528/39, 430/272.1, 525/477

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
Structure search conducted by STIC

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
Please See Continuation Sheet

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,E	US 6,268,457 (KENNEDY ET AL.) 31 July 2001 (31/07/01) Example 6	1-18

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

\* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

19 September 2001 (19.09.2001)

Date of mailing of the international search report

25 OCT 2001

Name and mailing address of the ISA/US

Commissioner of Patents and Trademarks  
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# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US01/22232

## Continuation of B. FIELDS SEARCHED Item 3:

EAST

anthracene methanol, naphthalene methanol, acetoxysilane, anthracene methoxy-methyldiethoxysilane, anti-reflective coating, ARC